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# CANADIAN PATENT

COMMINUTING ELASTOMERS

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This invention relates to a process for comminuting elastomers. More particularly it relates to a method of comminuting elastomers by which free-flowing particulate elastomer is obtained.

5 Both synthetic and natural rubber are supplied to users in the form of large pieces called bales. For many uses it is necessary to reduce the rubber to particulate form in order to subject it to processing. It has been proposed to reduce the rubber by working it in  
10 many kinds of apparatus variously called grinders, shredders, chippers, hogs, and so forth, which chop the rubber to small size chunks. A problem found to be associated with these reduction processes is that the small particles of rubber obtained from the apparatus  
15 are quite tacky and adherent, and are found to agglomerate or stick together forming large balls or masses of rubber which are not suitable for use in subsequent processing in such form. An attempt has been made to overcome this re-agglomeration of the rubber by feeding  
20 wood flour along with the rubber into the comminution apparatus. While this approach is found to prevent re-agglomeration of the rubber, the wood flour is very difficult to remove before further processing of the rubber. Ordinarily, a sizeable amount of wood flour is found to  
25 be present in products made from the so-treated rubber, constituting a contaminant therein and adversely affecting the properties of products made therefrom.

A primary object of the present invention is the provision of a process whereby elastomeric materials  
30 are obtained in free-flowing comminuted form. Another



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object of the present invention is the provision of a process in which non-agglomerating shredded rubber is obtained which does not contain contaminants which adversely affect the properties of products made therefrom. A further object of this invention is to provide a rubber grinding process which is economical and is easily adapted to existing equipment and procedures. Other objects and advantages of the present invention are obtained from the practice thereof as will become more clearly apparent.

In accordance with the present invention, the objects thereof are attained by feeding elastomeric material to a rubber reducing apparatus and simultaneously feeding a small amount of pulverized polyolefin into the reducing apparatus. The pulverized polyolefin is fed into the apparatus in such a manner that it is present in the zone at which the elastomeric material is engaged by the reducing mechanisms of the comminuting apparatus. Particulated rubber dusted with the powdered polyolefin is obtained in a non-agglomerating, easily handleable form for further processing.

The terms "rubber", "elastomer" and their derivative forms, as used throughout this specification, are employed in their usual meaning as understood in the arts, and include all rubbery materials, whether of natural or synthetic origin. These materials are characterized by being elastic and stretchable and by showing rapid retraction after release of the stretching force. They are polymeric and have molecular weights in excess of 1,000. The preferred polymers are linear in structure and may be saturated or unsaturated. Among the kinds

of rubbery materials that can be used to form the composition of this invention are polyisobutylene, butyl rubber, butadiene rubber, rubbery copolymers of butadiene with styrene, rubbery copolymers of butadiene with acrylonitrile, 5 rubbery copolymers of ethylene with propylene, isoprene rubber, whether natural such as India rubber and para rubber or synthetic such as polyisoprene, silicone rubber, polysulfide rubber (e.g. of the "Thiokol" type) and chlorinated and hydrochlorinated rubbers whether made from chlorine-containing monomers or by after-chlorination or hydrochlorination. 10

Polyolefins which can be used to produce the discrete particles of shredded rubber desired in the present novel process are solid resinous polymers of aliphatic 15  $\alpha$ -olefins having from 2 to 8 carbon atoms, such as ethylene, propylene, 1-butene, and 1-octene, whether homopolymers or copolymers of these monomers with each other. Representative examples of such polymers are polyethylene, polypropylene, copolymers of ethylene with propylene, copolymers of ethylene with 1-butene and so forth. These polyolefins preferably have molecular weights in excess of 20 about 4,000. Especially suitable for use in the process of this invention are essentially unbranched polymers of ethylene of intermediate or high density made by the so-called low pressure polymerization process using 25 Ziegler type catalysts.

Numerous advantages accrue to the process of this invention as a result of the use of powdered polyolefins to obtain particulated rubber. Several of these appear when 30 the particulated rubber is to be used for forming a homogeneous intimate blend of rubber with a polyolefin such as polyethylene, as by mixing the particulated rubber with pelletized polyethylene in mixing extruder or a Banbury mixer. One such advantage of the

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presently disclosed and claimed process is that the intimate blend contains only rubber and the polyolefin and is not contaminated with undesirable foreign matter. Foreign materials, such as the wood flour mentioned above and talcum powder, adversely effect the clarity of packaging films made from such blends. Another advantage of this process is that the shredded rubber made by this process can be used in connection with food and drugs when made into wrappings and bags after blending with polyolefins since the foods or drugs are not contacted with noxious materials. Still another advantage of the present invention is that it does not demand careful regulation of the amount of polyolefin used in the comminuting process as would be required when wood flour or other contaminating material is used. For example, when wood flour is employed in a rubber comminuting process it is necessary to use a sufficient quantity thereof to obtain nonself-adherent shredded rubber but the use of more than the required amount must scrupulously be avoided for the reason that the wood flour seriously impairs the clarity and general appearance of thin films when incorporated therein. When the herein disclosed process is used in comminuting rubber, the use of powdered polyolefin in slight excess is not harmful and, accordingly, careful regulation of the amount of polyolefin fed to the rubber grinder is not required.

Another advantage of the present invention is that while many dusting powders tried for the purpose of preventing coalescence of rubber crumbs tend to sink

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into and be enveloped by the substance of the rubber onto which they are coated, and some dusting agents become at least partially dissolved in the rubber, the polyolefin powder used in the process of the present invention does not suffer these difficulties. The polyolefin powders retain their effectiveness even at relatively high atmospheric temperatures which lower the viscosity of the rubbery material.

The polyolefin supplied to the shredded face of the rubber piece in the comminuting apparatus in accordance with this invention must be of small particle size. It has been found that the finer the powdered polyolefin, the more efficient is the process. Particle sizes smaller than 25 mesh, for example, within the range of 200 to 400 mesh, or even finer, find application in the present process. Such finely divided polyolefines can be obtained by any of various techniques. The polyolefin can, for example, be ground or ball milled, or treated in a colloid mill in order to pulverize the same. Suitable powders can also be obtained by spray drying a solution of a polyolefin. These techniques can be used alone or in combination with each other or with other methods understood in the art. Small amounts of powdered polyolefin have been found to be effective in rendering the comminuted rubber non-adherent. For example, feeding as little as 1/2 per cent by weight of polyolefin, based on the weight of the elastomer fed to the grinding apparatus, is suitable for producing free-flowing comminuted rubber. Feeding amounts of polyolefins in excess of about 10 per cent is of no particular advantage to obtaining a non-agglomerated product, while amounts in the range of about 3 per cent to 7 per cent are preferred in most instances.

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Throughout this specification parts and percentages are given and these are parts and percentages by weight.

EXAMPLE I

5 A 40-pound bale of polyisobutylene rubber, having an average molecular weight of 120,000, measuring approximately 8" x 14" x 14", is fed through the hopper guide of a Mitts and Merrill rubber hog to the bladed rotor turning at the rate of about 10,000 revolutions  
10 per minute. Two pounds of powdered polyethylene resin having a density of 0.935 gram per cubic centimeter, a Melt Index of 0.25 and a particle size of 325 mesh is fed slowly into the hopper and flows between the polyisobutylene bale and the hopper guide walls to the face  
15 of the bale engaged by the blades. The polyisobutylene is comminuted by the rotating blades and passes through a 1/4" mesh screen where it is collected. Substantially free-flowing, non-tacky comminuted polyisobutylene, dusted with a polyethylene powder, is removed from below  
20 the screen.

Forty-two parts of the product of Example I is dry blended in a rotating drum blender with 78 parts of polyethylene resin pellets having a density of 0.935 gram per cubic centimeter and a Melt Index of 0.25.  
25 This dry blend is passed through a mixing extruder in which it is fused and intimately mixed. The extrudate, a substantially homogeneous intimate blend containing 67 per cent polyethylene and 33 per cent rubber, is pelletized and collected. These pellets are fed to a screw extruder fitted with a long slit die and extruded as a  
30 film. The film is strong, suitable for use in food pack-

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aging, and of maximum clarity obtainable in a film of polyethylene and polyisobutylene rubber blended in this proportion.

5           The present invention may be effectively carried out with many variations of the above embodiment. For example, other kinds of reducing apparatus can be used. The size of the screen used to control the dimensions of the comminuted rubber particles can be varied. Further, additional equipment can be employed in conjunction with the reducer. For example, the hog can be  
10           equipped, below the screen, with a conduit and a blower for the swift transfer of the easily handled reduced material away from the hog to the next processing stage or to storage.



THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. A process of comminuting elastomeric material for the production of small particles thereof which comprises, presenting a large piece of elastomeric material to a comminuting device, feeding powdered resinous polyolefin into the device, comminuting the elastomeric material in the presence of the powdered polyolefin and recovering substantially free-flowing comminuted elastomeric material.

2. A process for comminuting rubber for the production of small particles thereof, which comprises, feeding a large piece of rubber to a comminuting device, supplying at least about 1/2 per cent based on the weight of the rubber of powdered resinous polyolefin to the device, comminuting the rubber in the presence of the powdered polyolefin and recovering substantially free-flowing comminuted rubber.

3. A process for comminuting rubber for the production of small particles thereof, which comprises, feeding a large piece of rubber to a comminuting device, supplying about 1/2 to 10 per cent by weight, based on the weight of the rubber, of a powdered resinous polyolefin to the device, comminuting the rubber in the presence of the powdered polyolefin and recovering substantially free-flowing comminuted rubber.

4. A process for comminuting rubber for the production of small particles thereof, which comprises, feeding a large piece of rubber to a comminuting device, supplying about 3 per cent to 7 per cent by weight,

5 based on the weight of the rubber, of powdered resinous polyolefin to the device, comminuting the rubber in the presence of the powdered polyolefin and recovering substantially non-agglomerated comminuted rubber.

5 5. A process for comminuting rubber for the production of small particles thereof, which comprises, feeding a large piece of rubber to a comminuting device, supplying at least about 1/2 per cent based on the weight of the rubber of a finely powdered resinous polymer of ethylene to the device, comminuting the rubber in the presence of the finely powdered polymer of ethylene and recovering substantially free-flowing comminuted rubber.

5 6. A process for comminuting rubber for the production of small particles thereof, which comprises, feeding a rubber bale to a comminuting device, supplying a small amount of powdered resinous ethylene polymer having a particle size smaller than 25 mesh to the device, comminuting the rubber in the presence of the powdered ethylene polymer and recovering substantially free-flowing comminuted rubber.

5 7. A process for comminuting rubber for the production of small particles thereof, which comprises, feeding a rubber bale to a comminuting device, supplying a small amount of a finely powdered resinous polymer of ethylene having a particle size smaller than 200 mesh to the device, comminuting the rubber in the presence of the finely powdered ethylene polymer and recovering substantially free-flowing comminuted rubber.

8. A process for comminuting rubber for the production of small particles thereof, which comprises, feeding a rubber bale to a comminuting device, supplying between 1/2 per cent and 10 per cent of powdered resinous ethylene polymer having a particle size smaller than 200 mesh to the device, comminuting the rubber in the presence of the powdered ethylene polymer and recovering substantially free-flowing comminuted rubber.

9. A process for comminuting rubber for the production of small particles thereof, which comprises, feeding a rubber bale to a comminuting device, supplying between 3 per cent and 7 per cent by weight, based upon the weight of the rubber of powdered resinous polymer of ethylene having a particle size between 200 mesh and 400 mesh, comminuting the rubber in the presence of the powdered polymer of ethylene and recovering substantially free-flowing non-agglomerating comminuted rubber.

10. A process for the production of comminuted rubber which comprises feeding a rubber bale to a comminuting device, supplying a small amount of an essentially unbranched resinous high density polymer of ethylene having a particle size smaller than 200 mesh to the device, comminuting the rubber in the presence of said ethylene polymer and recovering substantially non-tacky comminuted rubber.

11. A process for the production of small particles of polyisobutylene rubber which comprises feeding a bale of polyisobutylene rubber to a rubber hog, supplying about 5 per cent by weight based on the weight of the polyisobutylene of powdered high density

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resinous polyethylene, said polyethylene having a melt index of about 0.25 and a particle size of about 325 mesh, comminuting the rubber in said rubber hog in the presence of the powdered polyethylene whereby comminuted rubber coated with said polyethylene powder is obtained.

12. A process as claimed in claim 1, 2 or 3, wherein the comminuted elastomeric material obtained is blended with a polyolefin.